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1 April - 30 June 1965

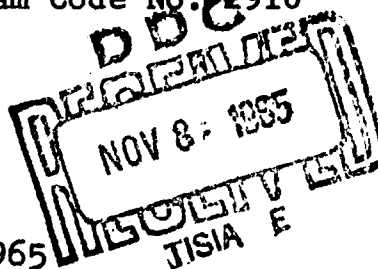
ELECTROCHEMICAL STUDIES IN THE SYNTHESIS
OF N-F COMPOUNDS

Contract No. Nonr 4054(00)

Research Project No. RR001-06-02

ARPA Order No. 399, Program Code No. 2910

October 27, 1965



TRACOR, INC.

1701 Guadalupe St. Austin, Texas 78701

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Prepared by:

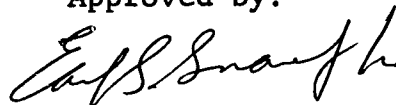
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Abstract

The electrolysis of 0.1M NH_4F in anhydrous HF at various anodic potentials has been studied during the past quarter with emphasis on the analysis of the volatile products formed. The major products formed in the gas phase were F_2 , cis- N_2F_2 and trans- N_2F_2 . The yield of both cis- and trans- N_2F_2 appears to increase with an increase in anodic bias.

Weight loss measurements and galvanostatic oxidation and reduction experiments were performed on nickel electrodes in HF at various water concentrations. Results indicate formation of a thick fluorine-saturated film on the electrode when anodically biased at +6.0 volts in anhydrous HF. Addition of water to the electrolyte inhibits corrosion and film formation.

ELECTROCHEMICAL STUDIES IN THE SYNTHESIS OF N-F COMPOUNDS

I. INTRODUCTION

Polarization curves obtained for the electrolysis of NH_4^+ in AHF are difficult to interpret accurately because of the large number of electrode reactions which occur. To aid in the interpretation of polarization curves, the recent work in this project has been devoted to determining qualitatively and quantitatively the products of electrolysis as a function of electrode potential from open circuit to +10.0 volts vs the $\text{Hg}/\text{Hg}_2\text{F}_2$ reference electrode. The major volatile products obtained from the electrolysis of NH_4^+ in AHF at potentials $> +6.0$ volts are F_2 , cis- N_2F_2 , and trans- N_2F_2 .

Experiments to establish further the conditions necessary for anodic passivation of metals in AHF have continued. This work is being pursued because of the large contribution of anodic current to electrode dissolution in AHF. Past work has shown that nickel electrodes can be anodically passivated in liquid AHF if 0.01% H_2O is added. This report gives the recent results obtained from weight loss measurements and galvanostatic redox experiments on nickel electrodes in HF containing $\leq 0.001\%$, 0.01%, and 0.10% H_2O .

II. EXPERIMENTAL APPARATUS

The only change made in experimental apparatus has been the use of two 7-foot, 1/4-inch o.d. Monel tubes (previously passivated with F_2) instead of one 16-foot Monel tube for the GC work. Only the carrier gas flows through one column, while the sample and carrier gas flow through the other column. Both of these columns are cooled and heated at the same rate in the same Dewar. This change was necessary to obtain a linear base line.

A diagram of the liquid IR cell that is being used for the liquid analysis of the electrolysis products is shown in Figure 1. The inlet line of the cell is connected to a line coming from the bottom of the electrolytic cell. A slight pressure on the solution inside the cell (i.e., a pressure increase due to volatile electrolysis products in the cell) can cause controllable amounts of the liquid to flow out of the bottom of the cell and into the IR cell without any change in state of the electrolytic solution. With this cell, liquid spectra of AHF plus soluble products can be observed between 800 and 5000 cm^{-1} . Dry ice and acetone mixture is used as the coolant in this work, although the cell can be cooled by other means such as liquid nitrogen. Variable Teflon spacers are available commercially for this type of cell. The cell can be sealed to hold at least 15 psi at -68°C . The only materials which the sample contacts are Teflon, Kel-F, Monel, and Irtran-2.

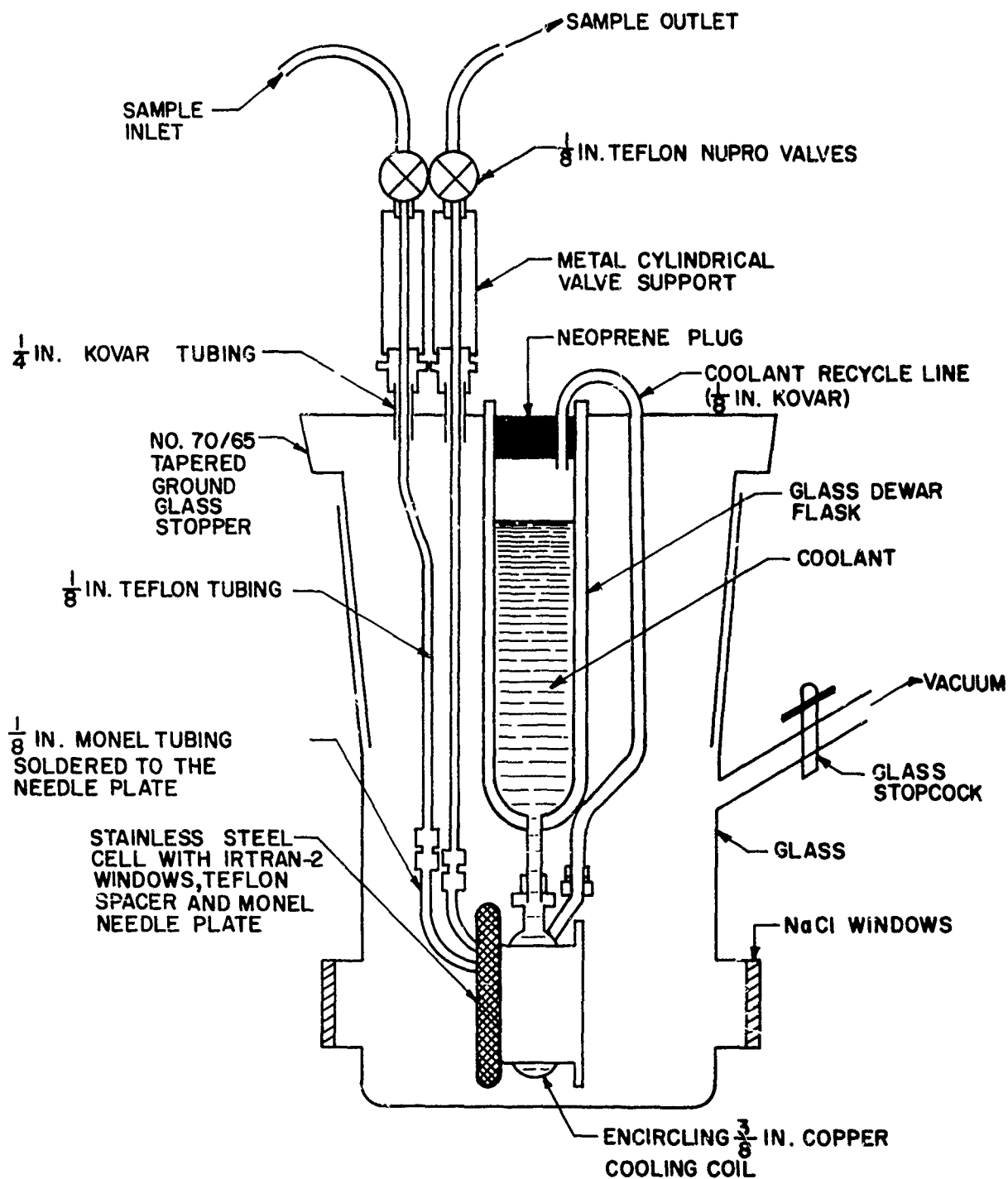


FIG. I- LOW TEMPERATURE INFRARED LIQUID CELL

III. ANALYSIS OF ELECTROLYSIS PRODUCTS

A major portion of the work this past quarter has been devoted to the analysis of the volatile electrolysis products by gas chromatography. An indication of the accuracy of the GC is shown in Table I. In this table, F_2 and HF are anhydrous gases obtained from Matheson, Inc., and the NF_3 , trans- N_2F_2 , and N_2F_4 are anhydrous gases obtained from Air Products, Inc. The same procedure was followed for these GC determinations as was given in the last quarterly report. However, the 40-foot column was replaced by a 7-foot column, and another 7-foot column was added for the reference flow of carrier gas. Both of the 1/4-inch Monel columns are contained in the same Dewar with each being exposed to the same temperature changes at the same time. By using both columns, a straight, reproducible base line was obtained.

The performance of this GC setup is shown in Table II. From these data, it is easy to see why the GC is so desirable for this type of analysis. The retention time is taken from the point at which the heating was started. The temperature of the column at this point is $-190^\circ C$. Due to the time between each peak, it is possible to isolate each of the volatile species at a reasonable purity so other types of analyses such as IR and mass spectra can be obtained.

Electrolysis of 0.1M NH_4F in AHF was performed at various voltages and the volatile products analyzed by the GC method discussed above. Results of these analyses are given in Table III. The F_2 yield decreases significantly with an increase in potential. Of particular interest is the effect of an increase in potential on the yield of cis- N_2F_2 . The yield of cis- N_2F_2 at +10.0 volts is almost 20 times greater than its yield at +6.0 volts. It is possible that one of the unknown products is OF_2 ; however, more IR data is necessary before the two unknown products can be

TABLE I

LOWER LIMITS OF DETECTION FOR POSSIBLE ELECTROLYSIS PRODUCT

<u>COMPOUND</u>	<u>LOWER LIMITS (ppm)</u>
F_2	210
NF_3	180
<u>trans-N_2F_2</u>	270
N_2F_4	250
HF	220

TABLE II

PERFORMANCE OF 7-FOOT MONEL COLUMN*

<u>COMPOUND</u>	<u>NO. OF THEORETICAL PLATES</u>	<u>COLUMN EFFICIENCY HETP (cm)</u>	<u>RETENTION TIME(min)</u>
F_2	0.64	334	0.2
NF_3	843	0.253	5.9
<u>trans-N_2F_2</u>	675	0.316	7.8
N_2F_4	537	0.396	11.6
HF	656	0.325	31.0

* Conditions: He flow = 50 cc/min, bridge current = 150 ma,
heating rate = 10°C/min between -190°C to -40°C.

TABLE III

GC ANALYSIS OF VOLATILE ELECTROLYSIS PRODUCTS^a

POTENTIAL	% F ₂	% cis-N ₂ F ₂	% trans-N ₂ F ₂	% NF ₃	% α ^b	% β ^b
+ 6.0 V	98.4	1.15	0.45	--	--	--
+ 8.0 V	86.71	6.15	6.79	0.09	0.13	0.13
+10.0 V	73.72	21.80	4.26	0.04	0.09	0.09

^aConditions: 1 cc sample, 50 cc/min He flow, 150 ma bridge current, 10°C/min program rate between -190°C to -40°C.

^bα and β = unknown products.

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identified. The dryness of the NH_4F -HF system used for these electrolyses is indicated by the absence of any significant amounts of volatile O-F compounds.

The gas and liquid IR spectra are still being interpreted and will be given in the next quarterly report.

IV. WORKING ELECTRODES

Cathodic stripping curves and weight loss measurements at +6.0 volts at various water concentrations were obtained on nickel electrodes in HF. Galvanostatic oxidation and reduction of nickel electrodes in HF solutions of various water concentration show the marked effect of water on film formation and character. Figure 2 shows galvanostatic oxidation and reduction curves for nickel in anhydrous HF. The anodic charging current of 15 ma/cm^2 was applied for six minutes with the potential reaching a value between 5.2 and 5.8 volts, well above the fluorine evolution potential. Immediately upon switching off the anodic current, a $10 \text{ } \mu\text{a/cm}^2$ cathodic current was applied to the nickel until a constant potential more negative than the open circuit potential was reached.

Figure 2 shows a potential arrest in the cathodic stripping region at about 2.5 volts, the approximate region of fluorine evolution. Upon removing the cathodic bias, the open circuit potential rose to a more positive value than the initial reading, indicating the persistence of a reducible species. Further application of cathodic current was required to attain the initial open circuit value. When this procedure was repeated (second run in Figure 2), the number of reduction coulombs involved in the potential arrest was about 2.5 times that required for a newly charged electrode.

The result is explained by the simultaneous formation of NiF_2 and fluorine on the electrode surface during the anodic charging. Application of the cathodic current reduces the fluorine contained in the film, leaving the electrode covered with NiF_2 . Subsequent anodic charging increases the thickness of the NiF_2 film, so a greater amount of fluorine can be trapped in the film, thus increasing the cathodic charge needed to reduce the fluorine. The reduction charge becomes constant only when the NiF_2 film (1) reaches a

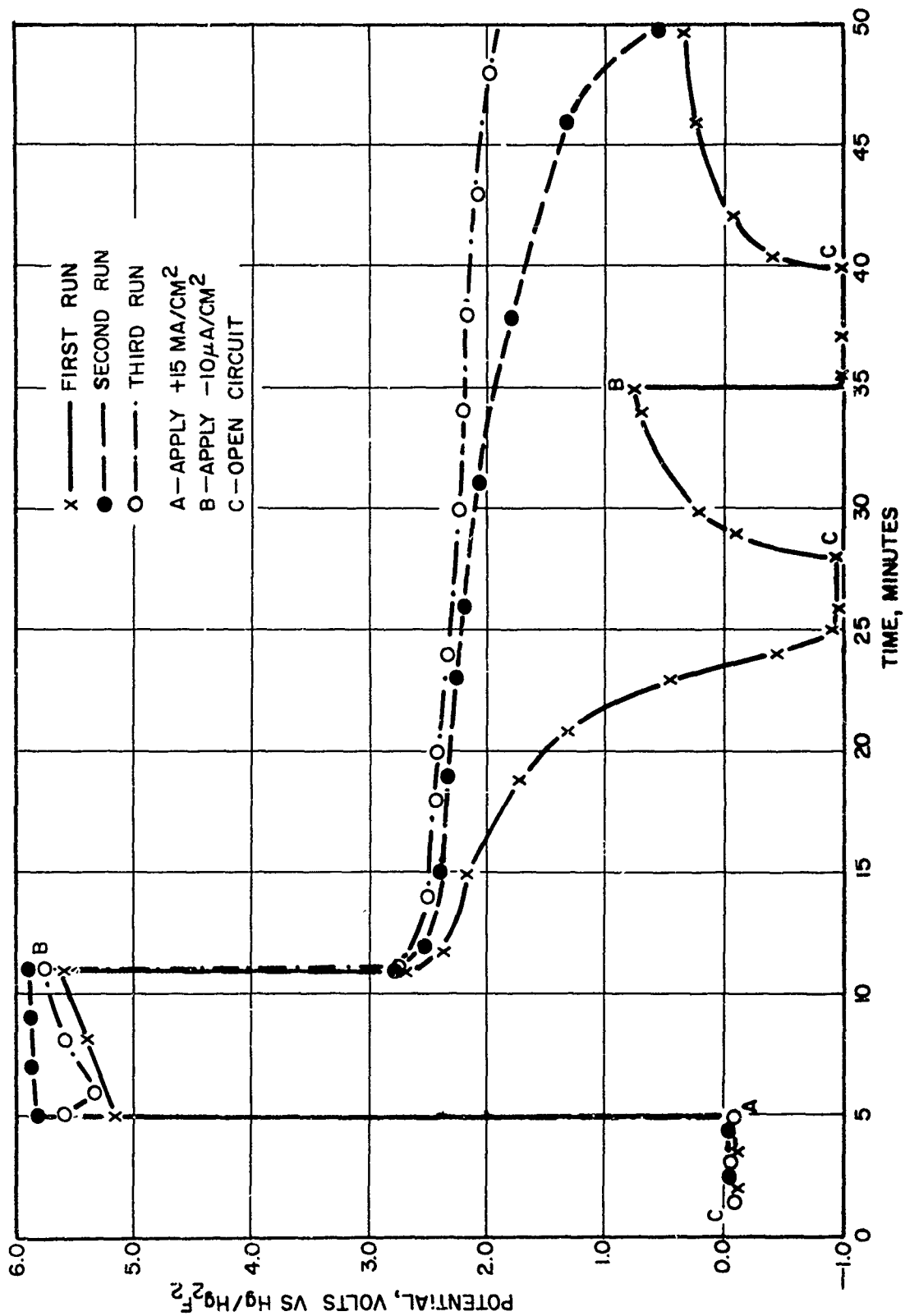


FIG. 2 — CATHODIC STRIPPING OF NICKEL IN $HF + 0.01M NaF + 10^{-3}\% H_2O$

limiting thickness and is saturated with fluorine during anodic charging or (2) is sufficiently thick that it is not saturated by the anodic charge. Weight loss data (see below) in anhydrous HF shows no limiting film thickness thus eliminating explanation (1). Confirmation of explanation (2) was obtained by removing the nickel electrode from the cell (in a nitrogen atmosphere) after anodic charging and applying a vacuum to the electrode. When placed back in the cell, these electrodes exhibited the original open circuit potential, and no potential arrest was observed during cathodic reduction.

Results of galvanostatic oxidation and reduction experiments on nickel electrodes in HF at various water concentrations are shown in Figures 3, 4, and 5. The potential arrest is reproducible after anodically charging the electrode twice in 0.01% H_2O (Figure 3), once in 0.02% H_2O (Figure 4), and very little arrest is observed in 0.1% H_2O (Figure 5), indicating an almost complete absence of bulk film formation.

Weight loss measurements were made on nickel electrodes at 6.8 volts in HF at various water concentrations. Figure 6 shows weight loss as a function of the anodic charged passed. Weight losses in anhydrous HF are quite high and show no evidence of passivation. Current efficiency for metal dissolution and film formation is about 70%. The addition of 0.01% water to the cell resulted in a decrease in the current efficiency for metal dissolution and film formation to about 2.6%. Weight losses at 0.10% water are even lower. These results are tabulated in Table IV. As shown in Figure 6, passivity of the electrode persists after passage of three times as much current as is needed to decompose all the water initially present. However, the HF solutions were not analyzed for water as electrolysis proceeded; and if the current efficiency for OF_2 , O_2 , and O_3 production were low enough, then an appreciable fraction of the water initially present could

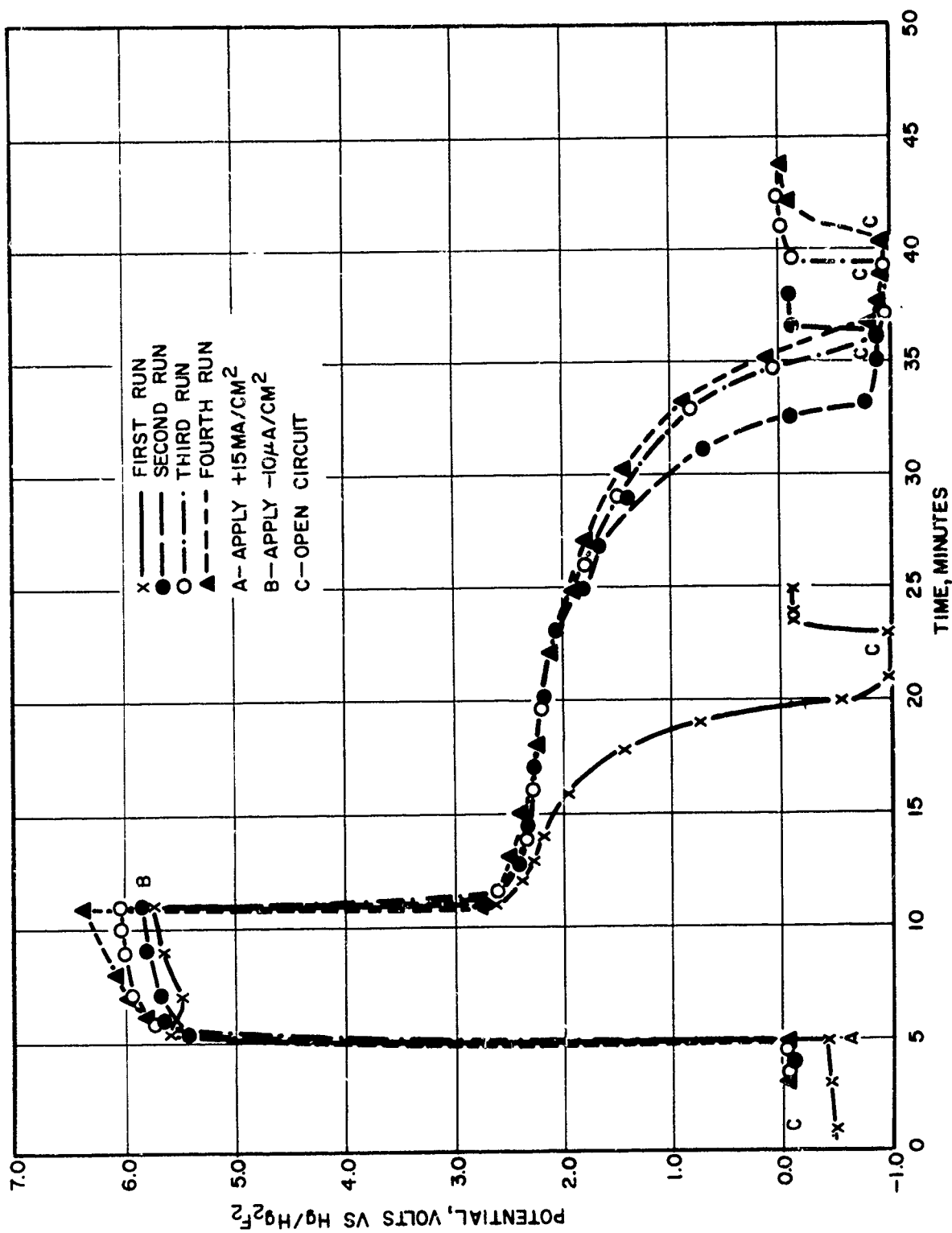


FIG. 3—CATHODIC STRIPPING OF NICKEL IN HF +0.01M NaF + 0.01% H₂O

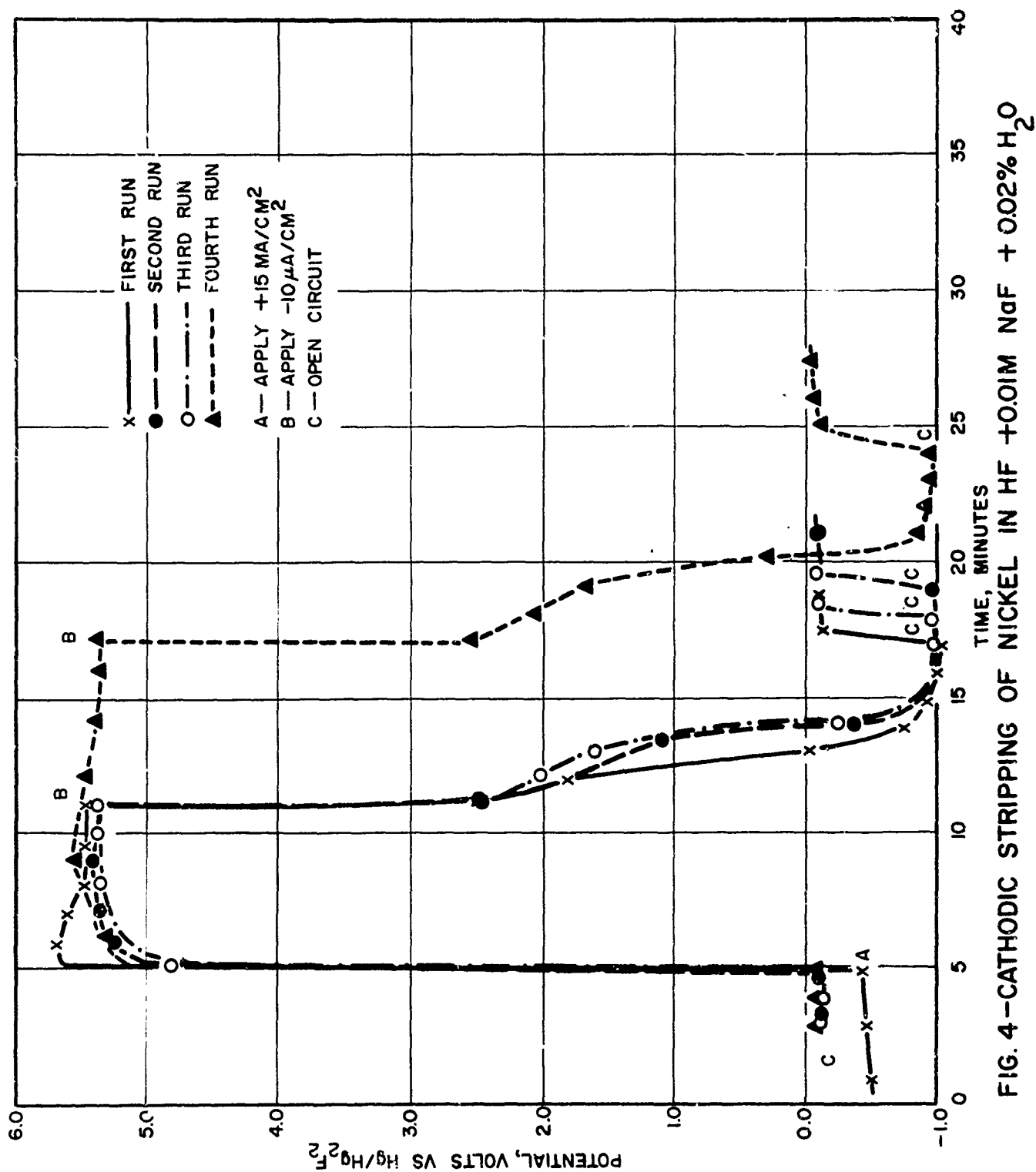


FIG. 4—CATHODIC STRIPPING OF NICKEL IN HF + 0.01M NaF + 0.02% H₂O

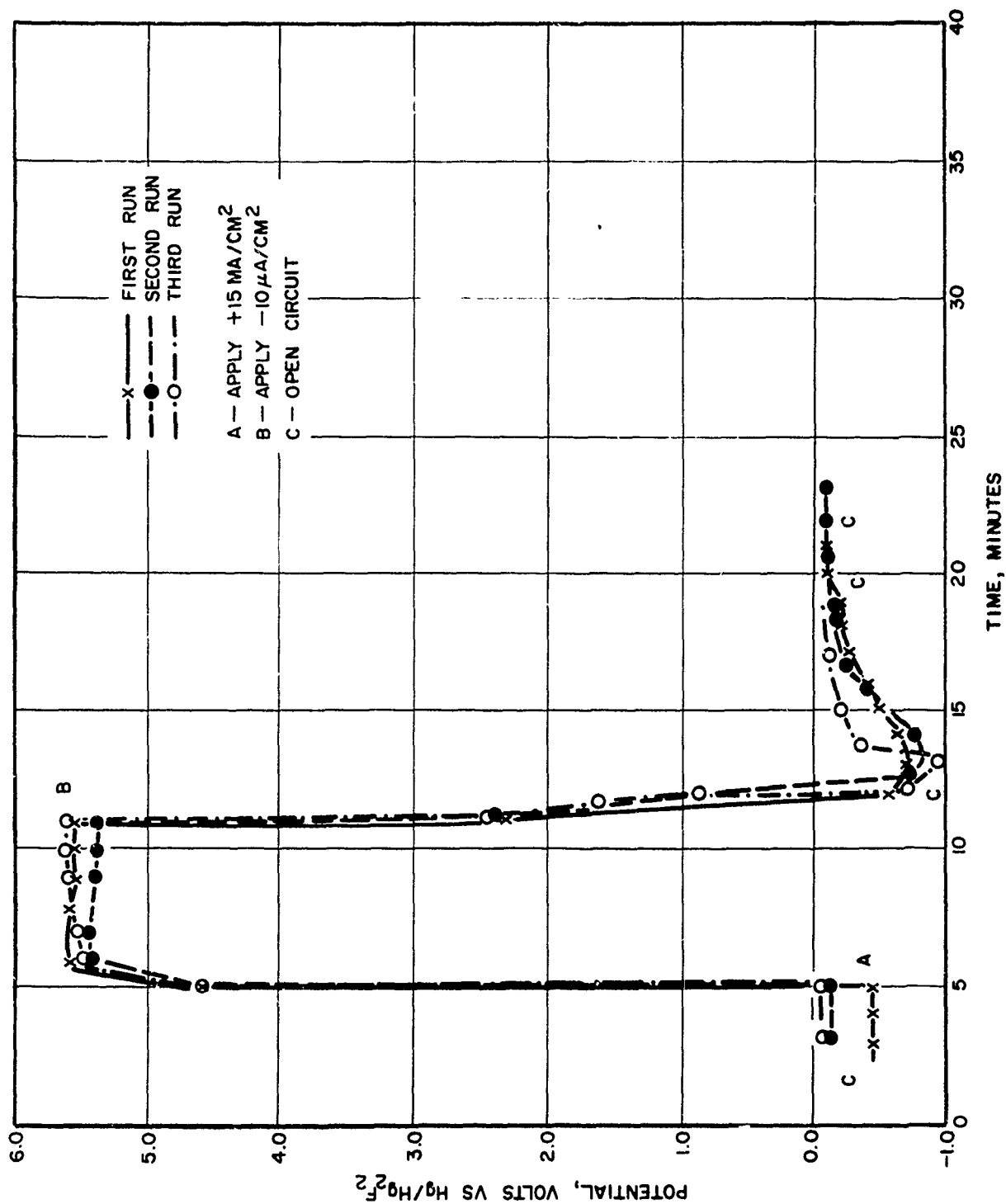


FIG. 5 — CATHODIC STRIPPING OF NICKEL IN HF +0.10M NaF +0.10% H_2O

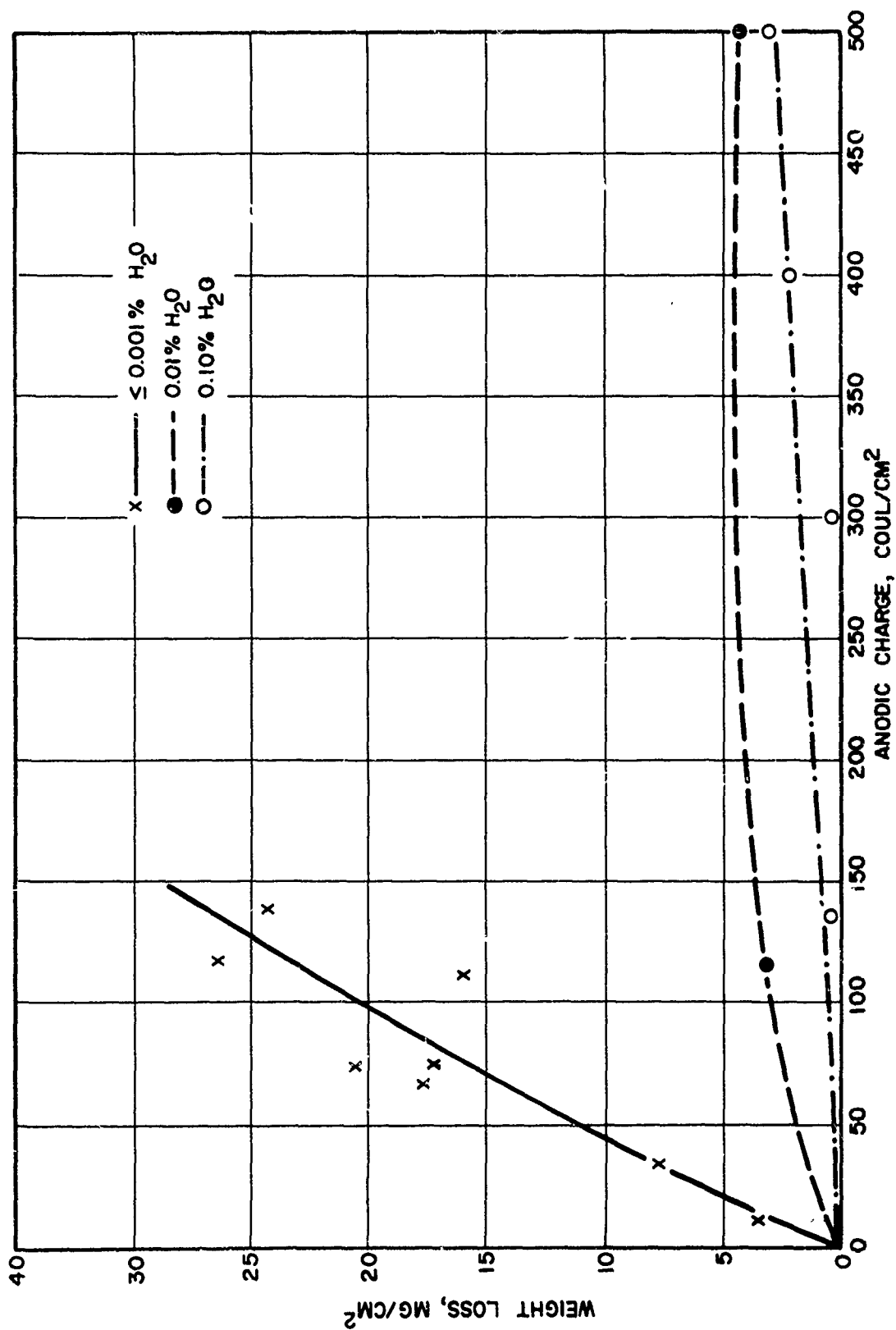


FIG. 6 - WEIGHT LOSSES ON NICKEL IN HF + 0.01M NaF + H₂O AT + 6.0V

TABLE IV

RATIO OF ANODIC CHARGE TO WEIGHT LOSS
FOR NICKEL IN HF AT +6.0 VOLTS

% H ₂ O	WEIGHT EQUIVALENT TO CHARGE (100 COUL)/WEIGHT LOSS
<0.001	1.5
0.01	10
0.10	50

have remained in the cell throughout the experiments. However, results obtained by Donahue and Nevitt* on electrolysis of wet HF show that OF_2 , O_2 , and O_3 account for about 90% of the current at a water concentration of 0.2%. Therefore, it is likely that most of the water initially present in these experiments was consumed during the longer runs.

Nickel is attacked at a high rate under anodic bias in anhydrous HF. As shown by the cathodic stripping curves (Figures 2 through 5), corrosion product (presumably NiF_2) forms under anodic bias and traps fluorine evolved from the electrode. It appears that the fluorine is held rather firmly in the film since an appreciable quantity is retained after subjecting the electrode to a vacuum for 15 minutes. As shown in Figure 7, an electrode was anodically biased to about +5.7 volts with a constant current of +15 ma/cm^2 applied at A. At D, the current was switched off and the potential fell to 2.8 volts (point C). The electrode was removed from the cell (under a nitrogen atmosphere) and placed in a vacuum for 15 minutes. After replacing the electrode in the cell (potential was 2.2 volts), a cathodic current of -10 $\mu\text{a/cm}^2$ was applied, resulting in the potential arrest, BE. This is similar to the potential arrest observed on electrodes not removed from the solution (see Figure 2). Using the same procedure, except for a 12-hour exposure to vacuum, eliminated the potential arrest, indicating that all reducible fluorine had been removed.

* J. A. Donahue and T. D. Nevitt, ARPA Propellant Contractors' Conference, Chicago, Illinois, April 13-15, 1964.

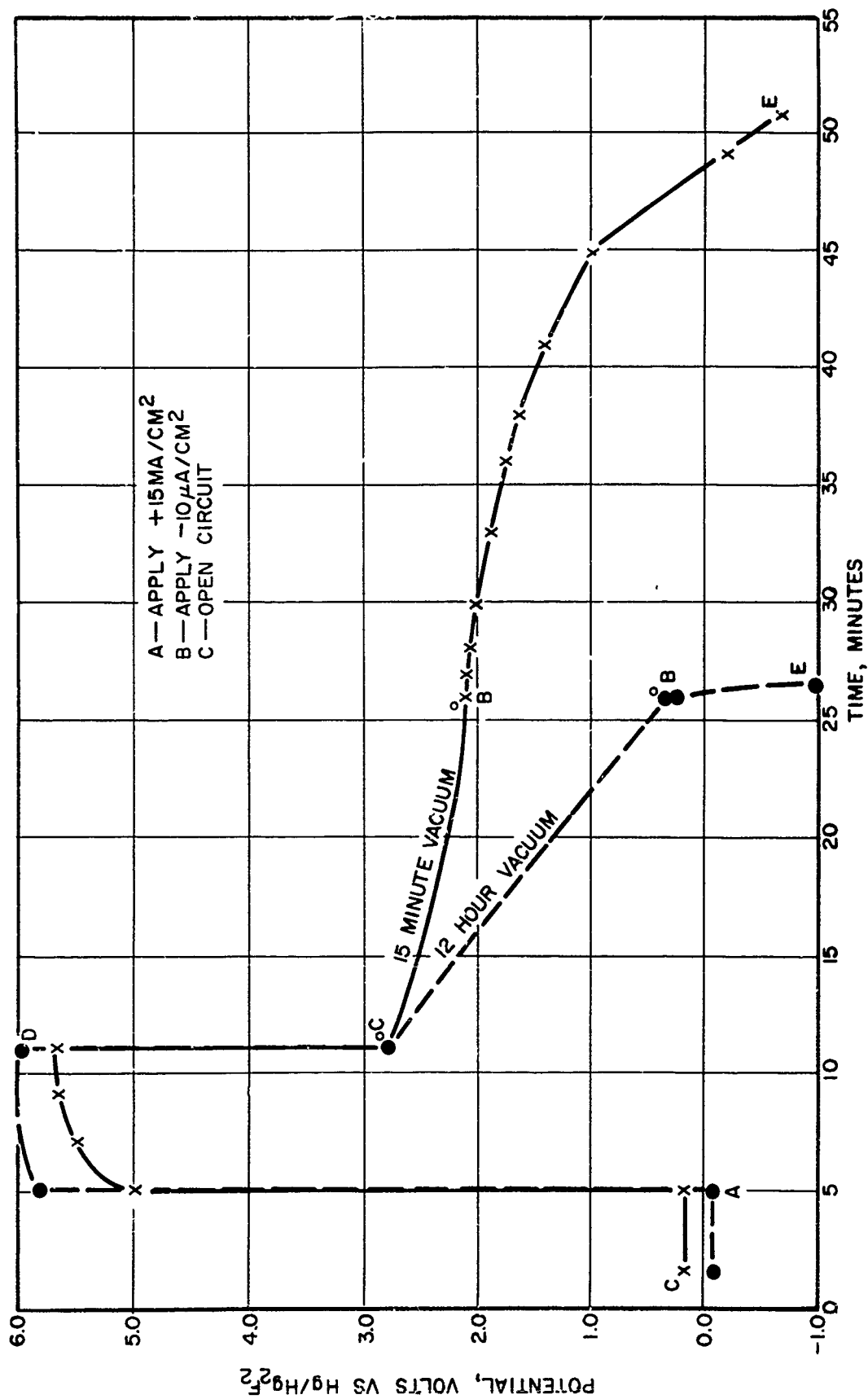


FIG. 7 --CATHODIC STRIPPING OF ANODICALLY POLARIZED NICKEL ELECTRODES AFTER EXPOSURE TO HIGH VACUUM

V. DISCUSSION

Analysis of the electrolysis products will be continued to obtain correlations between current passed and the amount of products formed. These data should help to elucidate the mechanism of electrochemical fluorinations in anhydrous HF. Chromatographic analyses are being supplemented by IR and mass spectrographic analyses of the liquid HF solution in the cell for possible direct observations of nitrogen-fluorine ionic species.

The utility of nickel as an anode for electrochemical fluorination in HF is markedly increased by the presence of traces of water (0.01%), which lessens the rate of attack on the electrode by an order of magnitude.

The use of passive electrodes for electrochemical fluorinations may result in a different spectrum of products. Electrodes used thus far (Monel) in anhydrous HF soon become covered with a thick metal fluoride film which is saturated with fluorine. Elimination of this type of film by the use of passive electrodes exposes the active solution species, NH_4^+ , to fluorine adsorbed on a metallic, electronic-conducting substrate. Thus, the different reaction conditions prevailing on passive and nonpassive electrodes may result in different fluorination mechanisms.

VI. FUTURE WORK

More electrolyses will be done using higher concentrations of NH_4F . Both the liquid and gas products will be analyzed by IR and GC. A potentiostat with a higher maximum output will be used to determine if there are any concentration-dependent plateaus past the fluorine evolution potential. The electrolysis of N_2H_5^+ in AHF will be studied in the same manner as that used for NH_4^+ . The effect of small amounts of water (added to the electrolytic solution) upon the electrolysis products will also be studied.

Electrical double layer capacity measurements will be made on nickel electrodes in HF at various water concentrations during the next quarter. X-ray and/or electron diffraction patterns of the various films which are formed on nickel will be obtained.

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